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SCIENTIFIC RESEARCH ON PAPAVER BRACTEATUM[1/]

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DETERMINATION OF THE THEBAINE CONTENT OF PAPAVER BRACTEATUM[2/]

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1/ General note: The views expressed by collaborating scientists in their contributions to the programme are not necessarily shared by the Secretariat.

2/ In the report of the Second Working Group on Papaver bracteatum (ST/SOA/SER.J/2), it was recommended that methods for the determination of thebaine in Papaver bracteatum should be compared and evaluated; to achieve this, it was decided that participants in the Working Group would forward their methods for publication in this series so that these could be tried out in the various laboratories.

3/ This paper was submitted by Mr. C. Candillon, Director of the Agricultural Department, Francopavot.

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The method used for separating thebaine from the various poppy alkaloids was based on one recommended by Pfeifer.[4/] This process enables thebaine to be isolated in the form of the tartrate.

I. Separation of the principal poppy alkaloids

The dry sample (roots, leaves or flowering tops) is reduced to a powder, which is ground in a mortar with a 5 per cent sodium carbonate solution. Five ml of alkaline solution are used for 10 g of dry powder.

After saturation of the powder, complete extraction of the alkaloids is effected by mixing with: methylene chloride:ether:methanol: 60:30:10 (V/V). Extraction continues for one hour. After filtration, the residue is extracted a second time. The extracts are combined and evaporated under reduced pressure. The dry residue is taken up with ether. This operation separates the ether-soluble pigments from the alkaloids, which are converted into salts by a solution of N/2 sulphuric acid. Separation is continued by the addition of aqueous ammonia (pH 7 to 8) to the aqueous acid phase. The alkaloid bases are then extracted with chloroform; 0.1 N sodium hydroxide is added to this organic phase, thus eliminating a few more pigments.

The alkaline phases have extracted morphine, narceine and narcotoline. These alkaloids were not detected in the various poppies studied: Papaver bracteatum and P. orientale. However, these basic solutions extract many pigments, thus justifying this procedure.

The thebaine base in chloroform solution is extracted by an 0.2 per cent tartaric acid solution. Under these conditions, codeine is also extracted in the form of tartrate, but the poppies studied only contain traces of codeine.

After collecting the aqueous acid phase, the thebaine base is extracted in chloroform in an ammoniacal medium. After separation and filtration through Whatman paper, the organic phase is dried under reduced pressure. The residue is subjected to chromatographic analysis and quantitative determination.

4/ S. Pfeifer, Bull. Narcot., 1958, X, No. 3, p.18.

II. Chromatographic analysis of the extract

The method of ascending chromatography on a thin layer of silica gel H is used. The solvent consists of the following mixture: toluene, acetone, methanol, 25 per cent ammonia: 45:45:7:3 (V/V). Migration takes place in 30 minutes. The solvent front is 10 cm.[5/]

The detecting agents are Dragendorff's reagent and concentrated sulphuric acid. When the latter is sprayed on the chromatogram thebaine appears as yellow-brown, while the other alkaloids remain invisible when cold, the colour being developed only after the plate has been placed in an oven at 100°C for 20 minutes.

Under these conditions, the thebaine has an Rf value of 0.45 to 0.50. Its amount as a proportion of the total alkaloid content is determined after spraying the chromatogram with Dragendorff's reagent and visual comparison of the thebaine spot with reference solutions for thebaine, developed under the same conditions.

Thebaine was found to be the main alkaloid in several samples analysed; on the other hand, a mixture of three or four alkaloids is very often found, in which thebaine represents only 30 to 50 per cent of the total alkaloid content.

III. Quantitative determination

The total quantity of alkaloids is determined by acidimetry.[6/] The results are expressed as the weight of thebaine per 100 g of dry plant.

Determination is carried out on the dry residue of the extract. The alkaloids are dissolved in a known excess of 0.02 N, say N ml hydrochloric acid, in the presence of methyl red. A solution of 0.02 N sodium hydroxide is added until the indicator turns yellow, say n ml.

Calculation of the results:

1 ml of 0.02 N sodium hydroxide corresponds to $\frac{311.3}{50}$ mg of thebaine

The test sample consists of 10 g of dry plant

Percentage of alkaloids (as thebaine): $\frac{(N-n) \times 311.3 \times 100}{10 \times 50 \times 1000}$

5/ Egon Stahl, Chromatographische und mikroskopische Analyse von Drogen.

6/ Pharmacopée française, VIIIe édition, p.167.

METHOD OF SEPARATING THE PRINCIPAL POPPY ALKALOIDS